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Validation of a sensor measuring suspended solids by attenuation of infrared light

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In this thesis, a real-time suspended solids sensor, designed and manufactured by a Finnish company Langis Oy, is tested, and validated. Testing was conducted in Metropolia Leiritie laboratories during April-June 2017. Water solutions with clay, potato starch, milk, and cement were measured with the sensor.

All the solutions yielded a satisfactory calibration curves; however, the calibration curves vary greatly between the different solutions. Hence, the readings of the sensor cannot be used as such to determine the concentration of suspended solids, but they are always depended on the chemical/physical composition of the water solution.

The sensor is at its best when it is used in circumstances, where the approximate composition of the water solution is already known, and the sensor is used for monitoring any changes in concentrations or maybe in composition – as this might be shown in change in readings.

Keywords	suspended solids, turbidity, sensors, waste water, process water, water monitoring, real-time monitoring

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Tässä insinöörityössä testattiin ja validoitiin reaaliaikainen kiintoaineanturi, jonka on suunnitellut ja valmistanut suomalainen Langis Oy. Testaus suoritettiin Metropolian Leiritien laboratoriotiloissa huhti-kesäkuun 2017 aikana. Anturilla mitattiin vesiliuoksia, joissa oli savea, perunatärkkelystä, maitoa ja sementtiä.

Kaikilla liuoksilla saatiin tyydyttävät kalibraatiokäyrät, mutta kalibraatiokäyrät vaihtelivat eri liuosten kesken. Tästä syystä anturin mittaustuloksia ei voi sellaisenaan käyttää kiintoainepitoisuuksien määrittelyyn, vaan ne riippuvat aina liuoksen kemikaalis-fysikaalisesta koostumuksesta.

Anturi onkin parhaimmillaan silloin, kun mitattavan liuoksen likimääräinen koostumus tiedetään, ja anturia käytetään mittamaan muutoksia kiintoainepitoisuuksissa ja mahdollisesti myös koostumuksessa, koska tämä saattaa näkyä mittaustuloksissa.

Avainsanat	kiintoaine, veden sameus, anturit, jätevesi, prosessivesi, vedenlaadun tarkkailu, reaaliaikainen mittaustarkkailu

Acknowledgements

Well, here we are! I'd like to thank Kaj and Erkki and also Antti Tohka for offering me some quite interesting opportunities.

Against all expectations, lab work turned out to be interesting and fun. For that I need to express my biggest thank-yous to the terrific trio of lab engineers Maru Åkerman, Marjut Haimila and Johanna Tikka for (often more or less hands-on) guiding. Cheers to you, ladies!

Contents

1 Introduction

One of the most important characteristics of the quality of water is the concentrations of suspended solids in it.

The composition of the solids can vary greatly, but nevertheless, the solids quantity itself is an important indicator of the quality of the water.

Suspended solids is a parameter that needs to be monitored, for example, in industrial process waters, natural waters, and waste water facilities.

Traditionally, monitoring of suspended solids is done by sampling and testing the sample in a laboratory. This causes delays in retrieving the results. As sensor and communication technologies advance, real-time monitoring becomes possible and sampling and testing can be skipped. Advantages of real-time monitoring are clear: changes in water quality can be detected immediately, which can be critical in some environments; water quality can be monitored continuously; and costs can be reduced by skipping sampling and testing.

In this thesis, a real-time suspended solids sensor meant for waste and process water monitoring is tested and validated. The testing was conducted in Metropolia Leiritie laboratories during April-June 2017.

2 Suspended solids

Solid material in water is either in dissolved or suspended form. Dissolved solids cannot be separated from the water by filtering. Suspended solids, on the other hand, can be filtered out.

Dissolved solids in water are mainly salts and other ions. Suspended solids are more variable material. The size and shape of the suspended solids particles can vary greatly. Suspended solids can be further divided in settleable and non-settleable solids. In still water, settleable solids settle down, but the settling time varies greatly, depending on the size, shape, and density of the particle. Non-settleable solids never settle down, but are permanently in suspense. These kinds of solids form emulsions in liquids.

The chemical composition of the solids can vary, and is important factor contributing to the quality of the water. The amount of suspended solids, however, can be used as such as a proxy of water quality. There are limit values for, for example, the amount of suspended solids in drinking water or effluent water from waste water treatment plants. In monitoring waste water or process water stream, changes in suspended solids levels indicate change in process conditions or other circumstance in the whole operational system.

3 Beer-Lambert

In water, the incident light is both reflected and absorbed. The absorbance of light is higher the more there are suspended solids in water. This is the base of the Beer-Lambert law.

Langis sensor measures the amount of suspended solids in water by emitting infrared light through a water-filled gap in the sensor and comparing the intensity of emitted light to the intensity of light detected on the other side of the gap. Suspended solids attenuate light going through the water volume in relation to the mass of the solids. The exact relation can be expressed as equation in Beer-Lambert law:

$$A = \log\left(\frac{I_0}{I}\right) = \varepsilon c,$$

which states that absorbance of the light intensity in the water volume is the logarithm of the fraction of light intensity entering the water volume and the light intensity leaving the water volume – the light intensity leaving being smaller, due to the absorbance. The law also states that absorbance is directly proportional to both the concentration *c*[1] of the substance in water measured and the path length *L* of the light, i.e. the distance between the emitter and the receiver of the light. In the sensor tested the distance cannot be altered; therefore, the term *L* can be omitted, and the equation can be re-written as

$$c = \log\left(\frac{I_0}{I}\right) \Big/ \varepsilon,$$

where new ε now includes also the term *L.*

That is, concentration of the particles in the water is directly related to the logarithm of the fraction of the ingoing/outgoing light.

[1] Concentration in mol units, that is. In empirical part of the thesis, the term “concentration” is used as in mg/l units. As it is explained in next chapter, these two magnitudes are closely related, but different.

4 Langis sensor

The sensor tested utilizes Beer-Lambert law to determine mass of suspended solids in water by calculating attenuation of infrared light in water with impurities. Beer-Lambert law states that attenuation is a function of concentration of those impurities. Concentration does not, however, equal mass, which also depends on density of the suspended material. Hence, the Beer-Lambert law can be only used as an approximation of the mass. With homogeneous material, or solutions with similar compositions, the density is roughly the same, and then mass roughly equals concentration. Hence, the measurements of the sensor cannot provide accurate values for mass.

A standard method of measuring the amount of suspended solids is filtering the sample and drying and weighing the residue that did not pass the filter (*SFS-EN 872. Water quality. Determination of suspended solids. Method by filtration through glass fibre filters.* 2005).

The method is time-consuming and requires professional equipment and lab personnel. With the sensor validated in this thesis, the amount of suspended solids is approximated/measured by turbidity measurements with the sensor alone. After installing and calibrating the device, minimal knowledge of laboratory work is needed.

While the sensor might well be not the as accurate as the standard method, it provides possibility for real-time measurements. In this thesis, it shown that in many environments, SS approximation with the sensor tested could be accurate enough for real-time measurements and monitoring. The accuracy of measurements is less important than possibility to detect changes in water quality when it happens.

Also, the possibility to monitor water quality remotely is very convenient for example in hazardous or inaccessible environments. Finally, as measurements can be saved automatically in database, comparing data over time is easy.

As with all continuously working sensors, wear and tear and fouling of sensor over time can lead to measurements change even though the water quality does not change. In Sections 6.6 and 7.4 some of the possible changes are addressed.

The device consists of sensor itself, which is an epoxy shaft (see Figure 1). On the end of the shaft, there is a gap that is immersed into water monitored. On one side of the

gap, there is infrared light lamp of 950 nm. On the other side of the gap, the detector detects light emitted. The difference between emitted and detected light energy is due to attenuation of the light due to suspended solids in water.

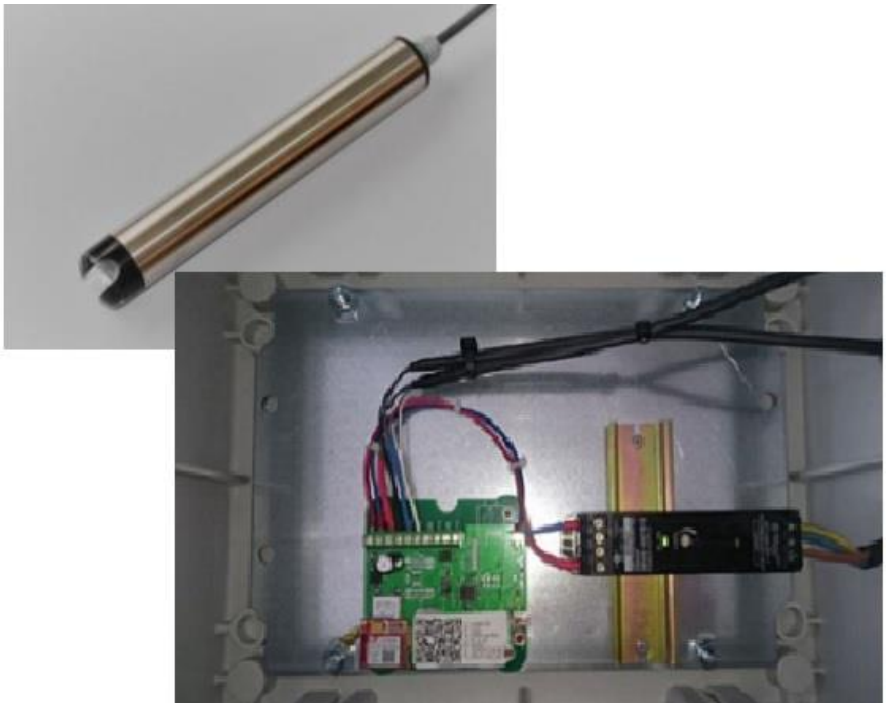

Figure 1. Sensor and its brain. At the bottom of the picture, the green circuit is the Arduino microcontroller. (modified from manual by Langis Oy)

The sensor is connected to the Arduino microcontroller with a slot for SIM card. Measurement data is sent to cloud service with the card. The data can be further accessed from the website or saved to the database.

5 Testing in lab

The sample, in a 100-ml beaker, was placed on a hot plate magnetic stirrer. The sensor was attached to a support stand with clamps so that it was immersed in the sample. (See Figure 2) The readings of the device for each concentration of the sample were recorded. The sample solution was diluted at ratio of 1:1 after recording. The device reports emitted and detected light in voltage and the ratio between them (0 to 1). This ratio was used to derive calibration curves.

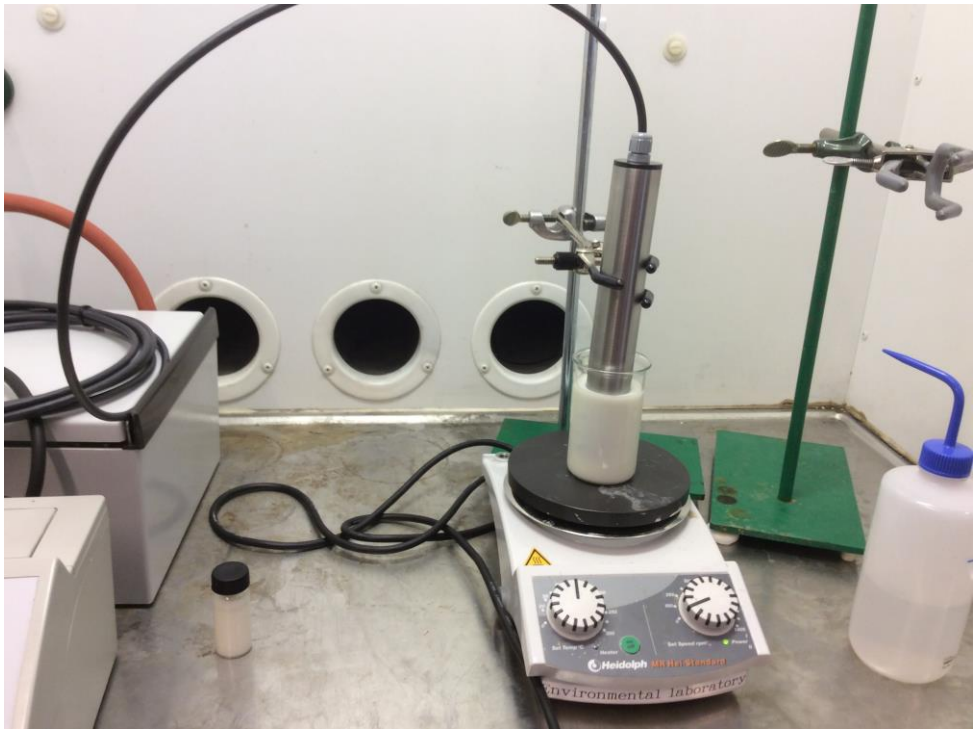

Figure 2. Testing set-up.

5.1 Solutions measured for calibration curves

Several water solutions – milk, starch, clay, and cement - were measured with the device. The solutions were both organic (milk, starch) and inorganic (clay, cement). Different solutions were used to observe the differences between readings given by the device.

Solutions used are presented in Table 1. Except for cement solution, dilutions were made by pipetting. Milk solutions did not need stirring when measured, but the other solutions needed to be stirred to prevent settling of the solids.

Clay, starch, and cement were weighed and mixed with 100 ml of water. The solutions were made separately for each day and run.

For milk, clay and starch solutions, dilution series were prepared by diluting the samples at a ratio of 1:1. Each dilution was done by pipetting half of the liquid (50 ml) from the beaker and inserting 50 ml of tap water to the beaker. The solution was mixed with a magnetic stirrer. With clay, starch, and cement, stirring was continuous, as otherwise solids would have settled during the measurements.

With cement, the dilution series was prepared beforehand, as pipetting the liquid was problematic, due to clogging of the pipette by sand in the cement mixture.

Part of the 50-ml pipetted from the solution was moved in a vial to measure the turbidity of the liquid. Turbidity measurements were compared with the measurements given by the sensor.

Tap water was used throughout testing, as the concentrations measured were relatively high, and any solids in tap water are mainly in dissolved form (e.g. (HSY 2017)).

Table 1. The solutions tested.

Component	Largest concentration, mg/l	Diluted by pipetting	Continuous stirring needed
Milk	40 100	X	
Milk	25 100	X	
Milk	27 600	X	
Clay	20 000	X	X
Clay	5000	X	X
Clay	34 200	X	X
Starch	150 000	X	X
Starch	34 200	X	X
Starch	30 000	X	X
Cement	40 000		X

5.1.1 Clay

A clay-containing soil sample from Southern Finland was sieved with standard sieves and four different fractions were obtained for use: particle size smaller than 63 µm, particle size between 63 and 125 µm, between 125 and 250 µm, and between 250 and 500 µm.

5.1.2 Milk

Three different types of milk were used: low-fat, lactose-containing milk (brand name Arki kevytmaitojuoma), fat-free but lactose-containing (brand name Arki rasvaton maitojuoma), and fat and lactose-free milk (brand name Eila rasvaton maitojuoma, laktoositon). The suspended solids concentrations of these milks were 40 100 mg/l, 25 100 mg/l, and 27 600 mg/l, respectively. Milk types used are presented in Table 2. Milks were obtained from Metropolia campus cafeteria.

Table 2. Milks tested.

	Arki kevytmaitojuoma: low-fat, lactose-containing	Arki rasvaton maitojuoma: fat-free, lactose-containing	Eila rasvaton maitojuoma, laktoositon: fat and lactose-free
Fat (insoluble)	1.5	0	0
Sugars (soluble)	4.8	4.9	3.1
Proteins	3	3	3.3
80 % of which insoluble caseins	2.4	2.4	2.64
Salt	.1	.1	.1
Calcium	.11	.11	.12
Together insoluble	4.01	2.51	2.76

The calculated values in Table 2 are based on the following aspects. Sugars in cow milk consist mainly of lactose, which is, in concentrations that are found in milk, water soluble (Machado, Coutinho et al. 2000, National Center for Biotechnology Information). Small amounts of free glucose and galactose can also be found in milk (Hurley 2010a). These are soluble in water in large concentrations (National Center for Biotechnology Information, Royal Society of Chemistry 2015).

Proteins in milk are mostly caseins, which are present in milk as casein micelles. The casein micelles are not soluble, but can be separated from the solution by centrifugation. Minor part of the proteins, whey proteins, are more soluble (Hurley 2010b). Approximately 80 % of milk proteins are in casein micelle form and hence insoluble (Anonymous 2017).

Calcium in milk is mostly trapped in the casein micelles, and hence insoluble, whereas other salts are mostly soluble (Hurley 2010a).

5.1.3 Starch

Starch was regular potato flour for cooking purposes (brand name Kolmen konstin perunajauhot) with a density of approximately 70 g/ 100 ml.

5.1.4 Portland cement

Quick-setting cement mix that includes sand was used for cement solutions.

With cement, the dilution series was prepared beforehand by mixing certain amounts of cement to 100 ml of water, as diluting during the run was not possible: when pipetting, the pipettes tend to clog with sand grains in the cement mixture.

5.2 Temperature

Responses for milk solutions at different temperatures (cold, room temperature, warm) were measured. Other solutions were tested in room temperature (around 22 ºC).

One test-run was also conducted with cold, room temperature, and warm tap water

To gain a more accurate information of the relation of temperature and the responses of the device, the temperature of tap water, with the sensor immersed, was measured with Vernier LabQuest device with temperature sensor. The temperature was measured over period of three days, with one minute’s interval, and the device’s responses recorded.

5.3 Data acquisition

During the first test runs, results uploaded continuously from the sensor onto web page were noted down – one response per each step and each concentration.

During later tests in June, a database was available. The data was automatically added to Mongo database, and was further processed with R and Excel. The average response of five minutes was calculated for each step and each sample.

6 Results

6.1 Calibration curves

One object of the thesis was to find calibration curves for different solutions. Apart from the cement solution, this was achieved.

As the device uses 11 different intensities of IR light for measuring, 11 calibration curves were obtained from each test run. As the calibration curves for individual intensities were quite similar (but see Section 6.5), and as it would not be illustrative to show each of the calibration curves separately, the responses from all the 11 steps (or intensities) were averaged to obtain single calibration curve for each test run. If not otherwise stated, the calibration curves below are averaged over the steps, but the different runs are shown separately.

6.1.1 Calibration curves for milk

There were three different kinds of milks tested, of which lactose-containing low-fat milk generated very similar calibration curves on different runs, but the calibration curves of lactose-free milk varied somewhat between the runs. Fat-free, lactose-containing milk was only used in one run. In Figure 3, all calibration curves are shown.

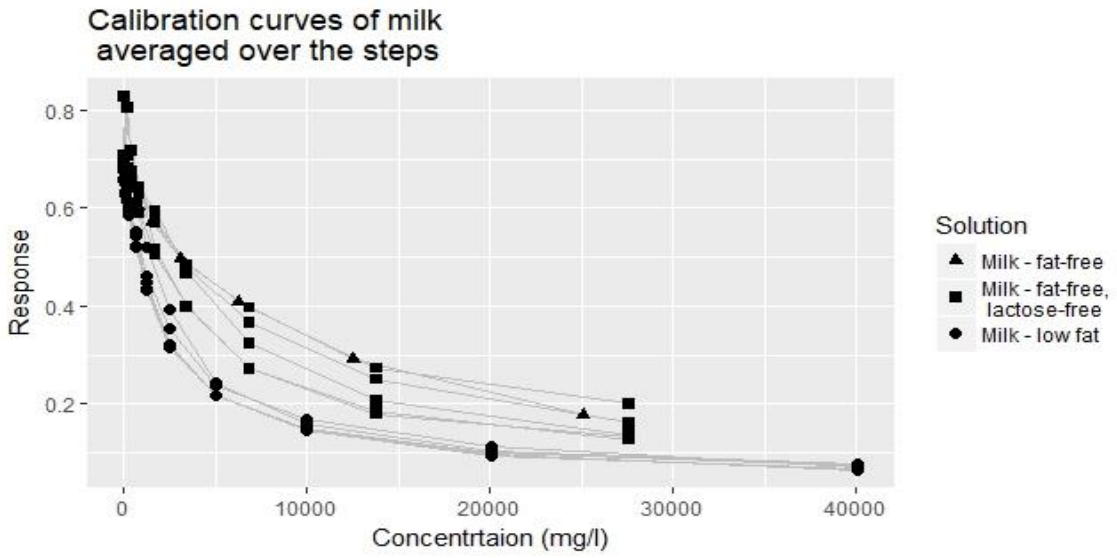

Figure 3. Calibration curves for all milk measurements. While low-fat milk dilutions provide quite consistent calibration curves, fat-free, lactose-free milk dilutions do not.

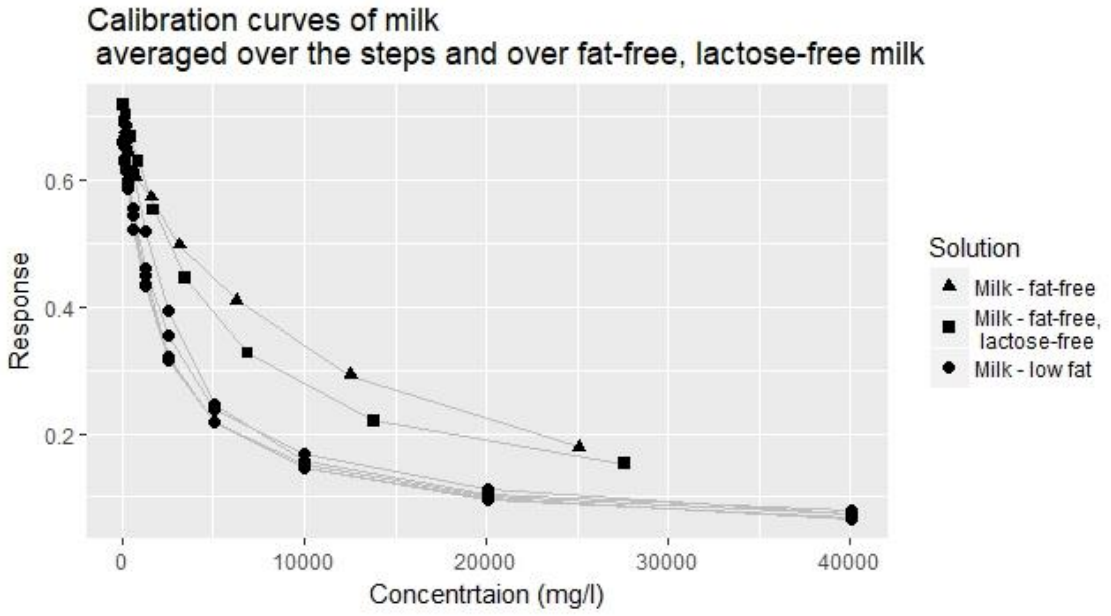

Figure 4. Averaged calibration curves for milk.

When the measurements for lactose-free-milk are averaged over the runs, the calibration curves for different kinds of milks are quite clear. For low-fat, lactose-containing milk, the five test-runs provide very similar calibration curves. The calibration curve for the average of lactose-free milk and the calibration curve for fat-free, lactose-containing milk are clearly different. (See Figure 4).

6.1.2 Calibration curves for starch

For starch, the calibration curve did not level out until quite high concentrations, and only one measurement run was conducted at that high concentrations. The three other runs, conducted with less concentrated solutions, gave, nevertheless, similar responses in lower concentrations, as can be seen in Figure 5.

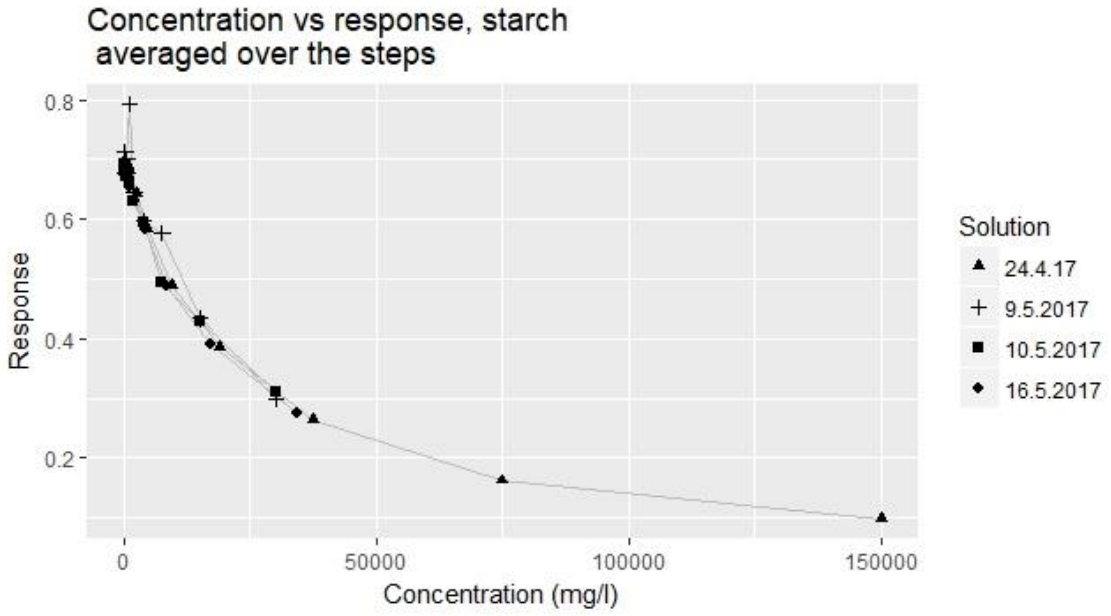

Figure 5. Calibration curves for starch. The measurements agree well between different runs

6.1.3 Calibration curves for Clay

Clay was initially sieved and separated into four different fractions, but the calibration curves of them were so similar (See Figure 6) that the test run 25.4. was averaged over the different fractions.

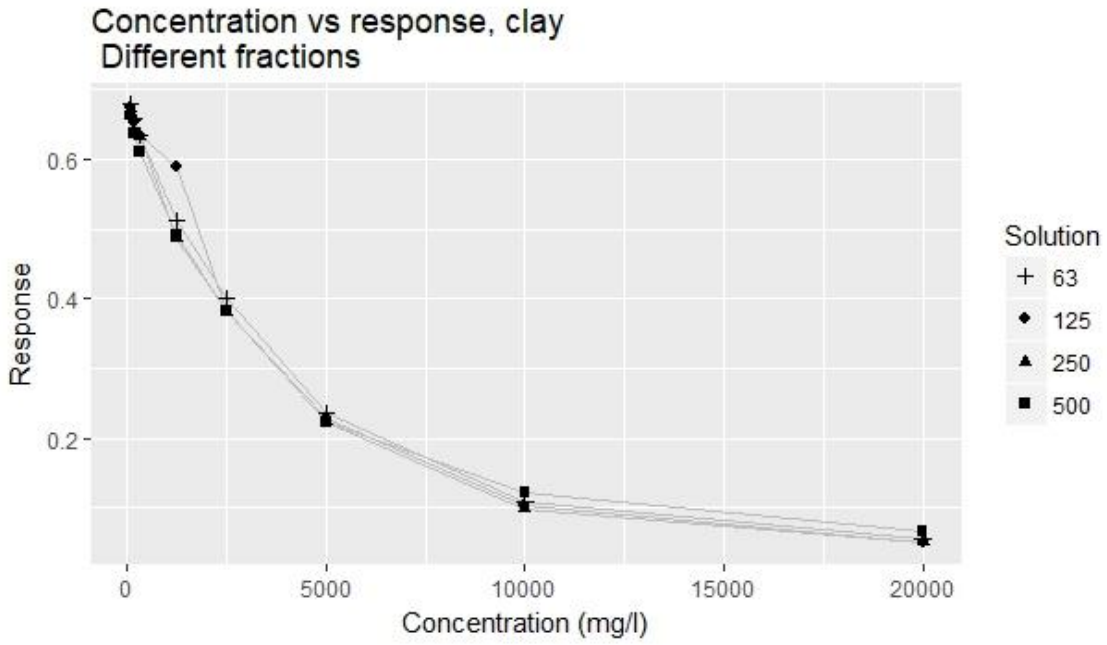

Figure 6. Even though clay was sieved to obtain different coarsenesses, they yielded very similar calibration curves.

In later tests, only fraction of grain size finer than 63 µm was used. In Figure 7, calibration curves for four different test runs (the first being averaged over the four different clay fractions, shown in Figure 6) are presented.

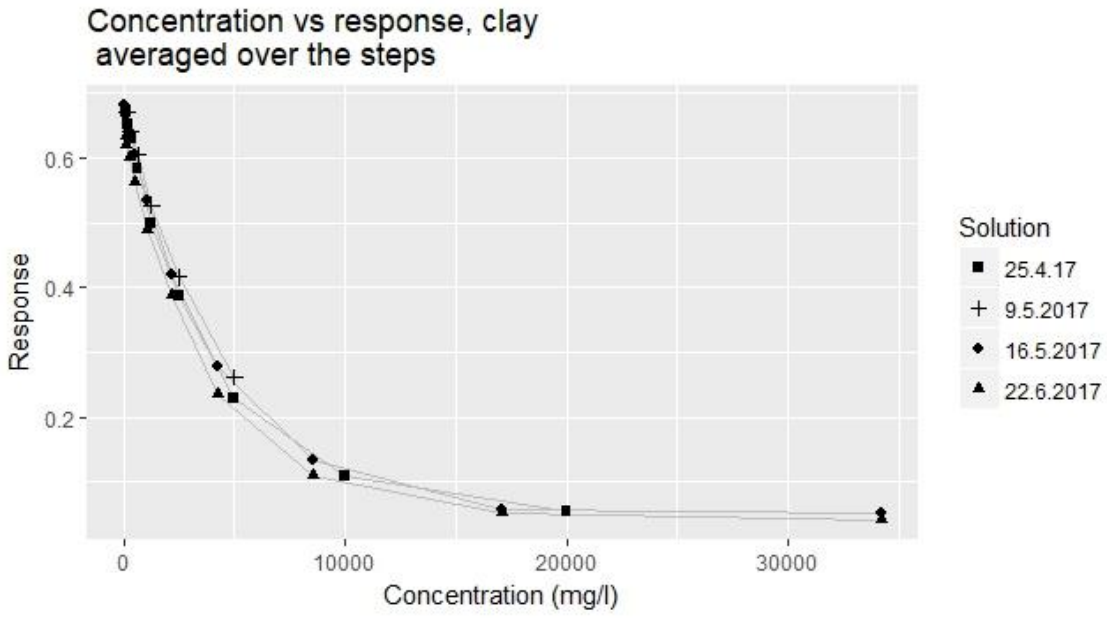

Figure 7. Calibration curves for clay on four different runs.

As can be seen in Figure 7, the calibration curves for four different test runs were quite similar.

6.1.4 Calibration curve for Cement

While the fitted curve for responses for cement solutions is strictly decreasing, it is not nearly as smooth as the curves for other solution. Also, the curve does not show the logarithmic shape expected (see Figure 8).

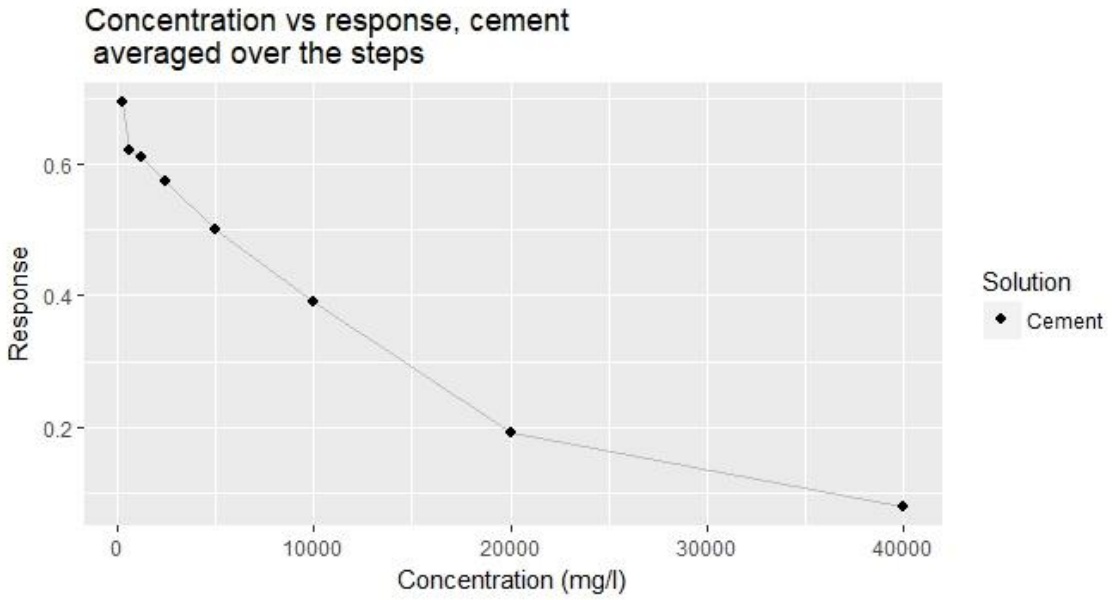

Figure 8. The calibration curve for cement is not as smooth as for some other solutions.

6.2 Different solutions, different calibration curves and different turbidities

While calibration curves for solutions with the same contents (especially for clay and starch) were quite similar on different runs, they were very dissimilar between solutions of different compositions. The response for starch, for example, was higher than for other solutions in all concentrations. (see Figure 9).

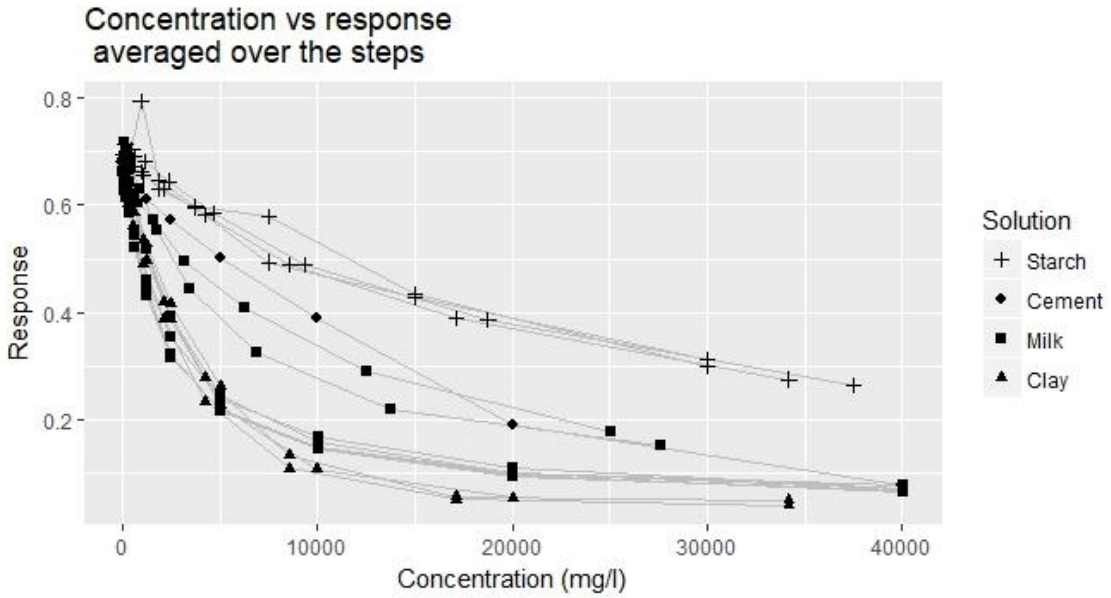

Figure 9. With similar or same concentrations of suspended solids, solutions with different compositions have very different calibration curves.

This is consistent with the fact that turbidity (FAU) values for starch solution were lower than for other solutions (except for cement, see Figure 10).

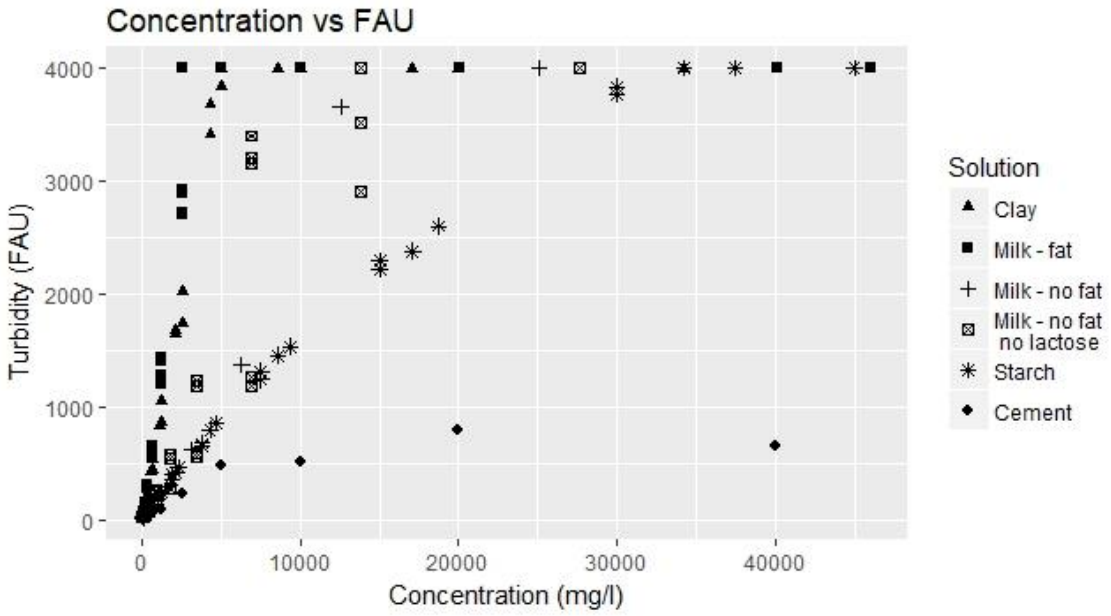

Figure 10. With same amount of suspended solids in the solution, turbidity values differ between different compositions of the solids. Solutions with clay or fat-containing milk seemed to be more turbid than starch or fat-free milk. Turbidity measurements for cement were also low, but they are not reliable, as without stirring, the solids in the solution settled too rapidly for proper turbidity measuring.

Also, between different milks there were differences in responses. The different kinds of milks varied in fat and lactose contents, and this was shown in responses, even though the concentrations of suspended solids were supposedly the same.

6.3 Temperature

Response for warm or hot or warm solution (milk) was smaller than for cooler solution. In each individual test run for temperatures the differences between different temperatures were clear. The differences were not, however, as clear in between-the-runs comparisons.

One run was conducted with water in different temperatures. In Figure 11 it is shown how the responses differed to hot water than to water in room temperature. Hot water yielded much lower responses with all measured steps.

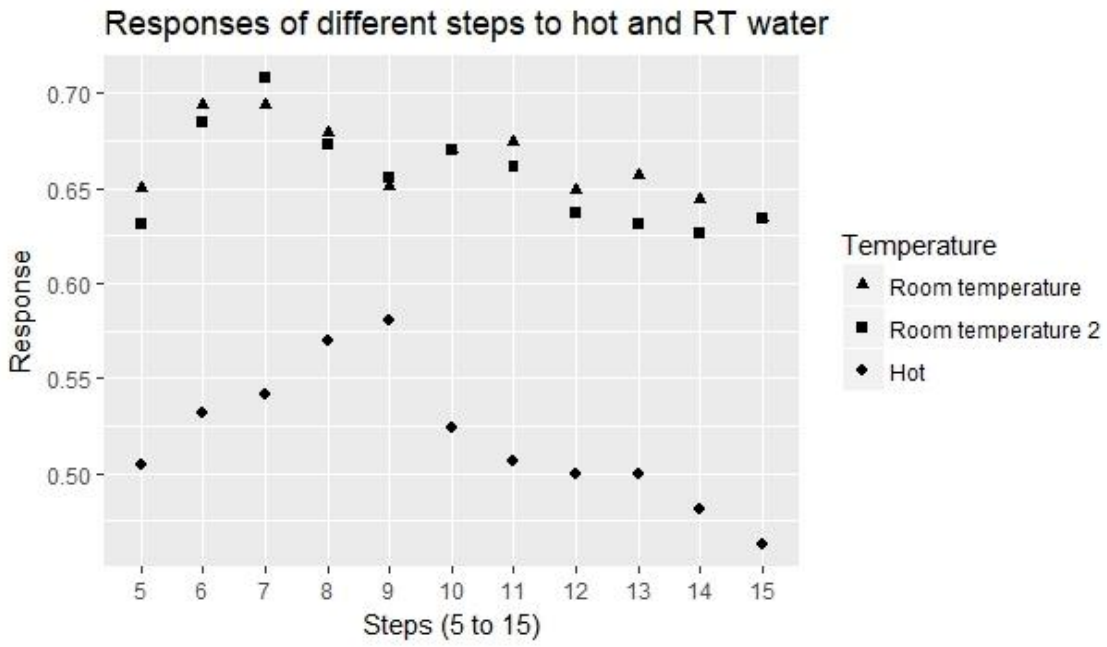

Figure 11. When responses to tap water were measured, there was small variation between two runs with water in room temperature, but the responses to hot water were clearly lower by all steps.

When the temperature of tap water and the responses from three days' time were compared, there was small but statistically very significant negative correlation between the variables: adjusted R^2 was 0.3356 and $p << 0.001$.

6.4 Air bubbles

As can be seen in Figure 12, adding air bubbles (in form of carbonated water) to milk solution interfered with the infrared light passing the solution, at least with some of the concentrations, and consequently, the response of the device was lower. Amount of the carbonated water did not affect – or sometimes affected in wrong direction - the readings of the device in these measurements.

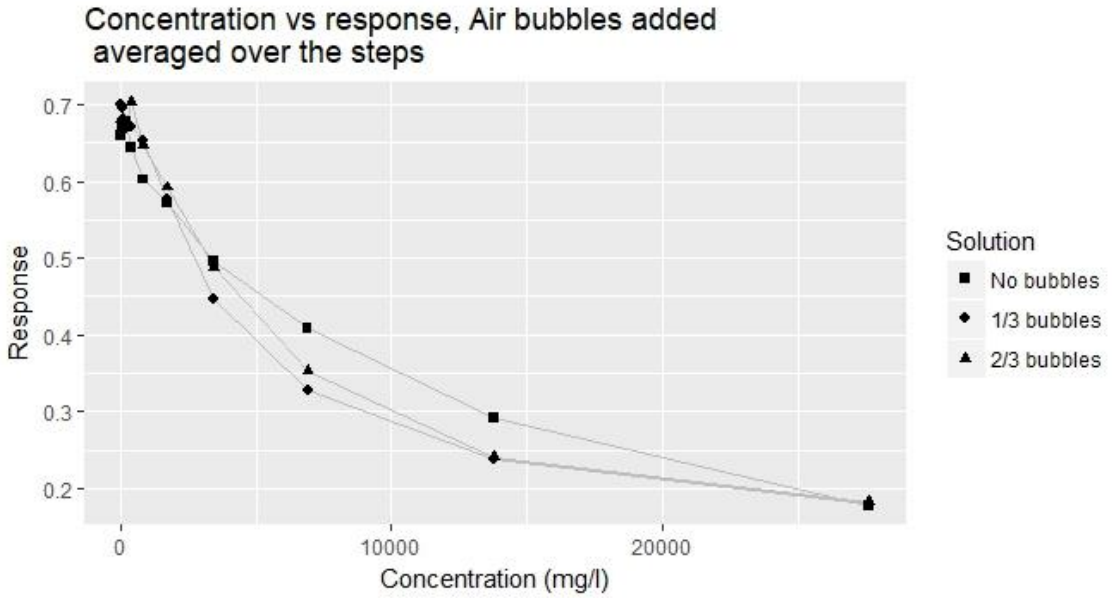

Figure 12. Solution without air bubbles yielded a higher response.

6.5 Differences between different step responses

Measurements with 11 different infrared light intensities resulted in similar responses and calibration curves. A typical set of calibration curves from one run is shown in Figure 13.

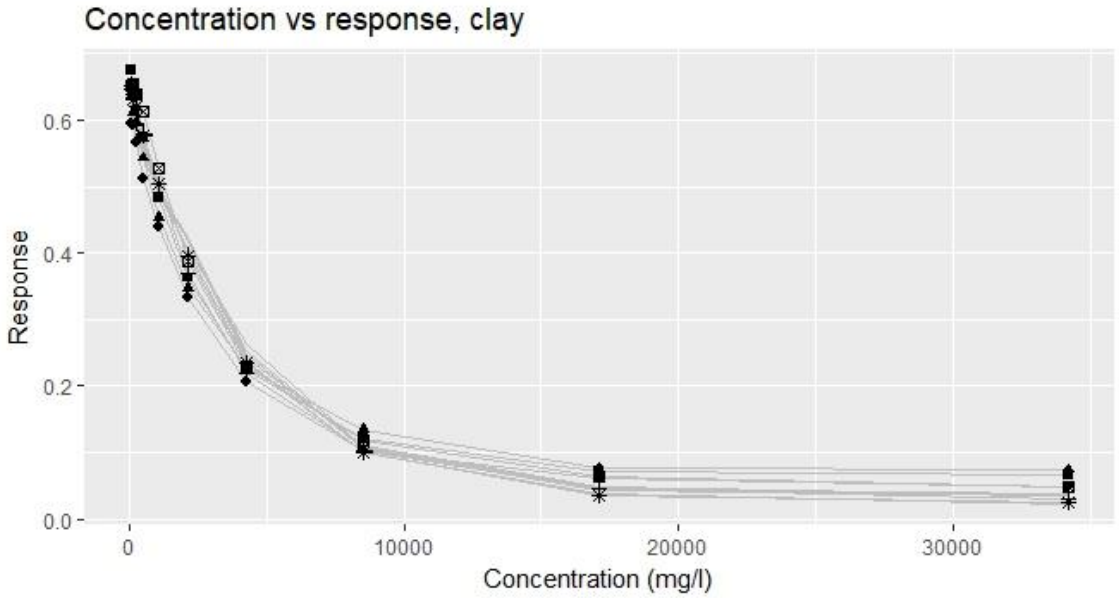

Figure 13. Different intensities (steps) yielded quite similar responses.

Especially the shapes of the calibration curves are reminiscent. Consequently, in figures with other results, the responses are averaged over the different intensities (steps).

On the other hand, relative differences in high concentrations were large. In large concentrations, also the relative variations were large, and the sensitivity of the sensor is very low. In large concentrations, the limitations of Beer-Lambert law are apparent.

When continuous measurements for three days (with water) were studied, differences between the step responses where, however, clear. In Figure 14. the responses are shown as a box and whiskers plot. The box represents each step's responses between the first and third quartiles, while the horizontal line in the box represents the median of the values. The whiskers (vertical lines) show responses with values that are outside the first-third quartile box, but lie within the distance of 1.5 times of the height of the box. The dots represent outliers.

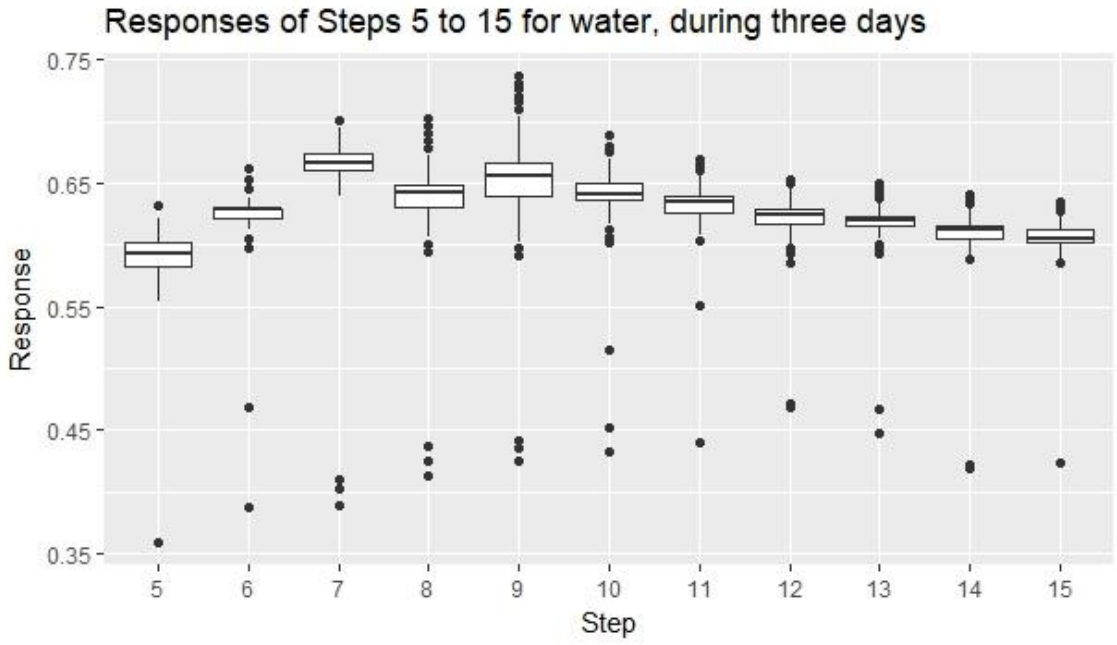

Figure 14. Even though is seems that there are no significant differences between responses from different steps when looked at the calibration curves, differences are clear when measuring for a longer period.

For some reason, outliers on lower side are a lot further from the median than outliers on higher side. This can be due to air bubbles or some other disturbance.

6.6 Decreasing responses

The device's response when no liquid was present (device was in air) were recorded during several test runs. In Figure 15 it can be seen that the responses, on the average, declined over time. [2]

[2] As there was only one measurement noted down per test run, there is some uncertainty in the values.

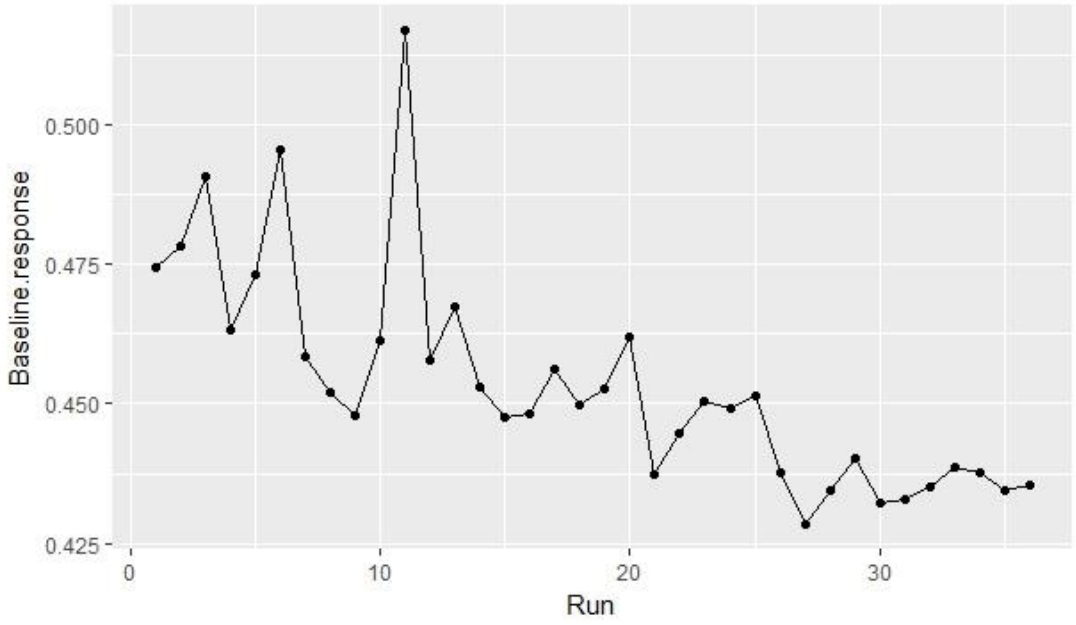

Figure 15. Baselines measured during April-May test runs showed declining tendency.

An interesting exception was baseline on run 11, which yielded the highest response. In this and previous runs, clay solution was used.

When measurements for tap water were recorded for three days, the decreasing tendency was also clear (see Figure 16).

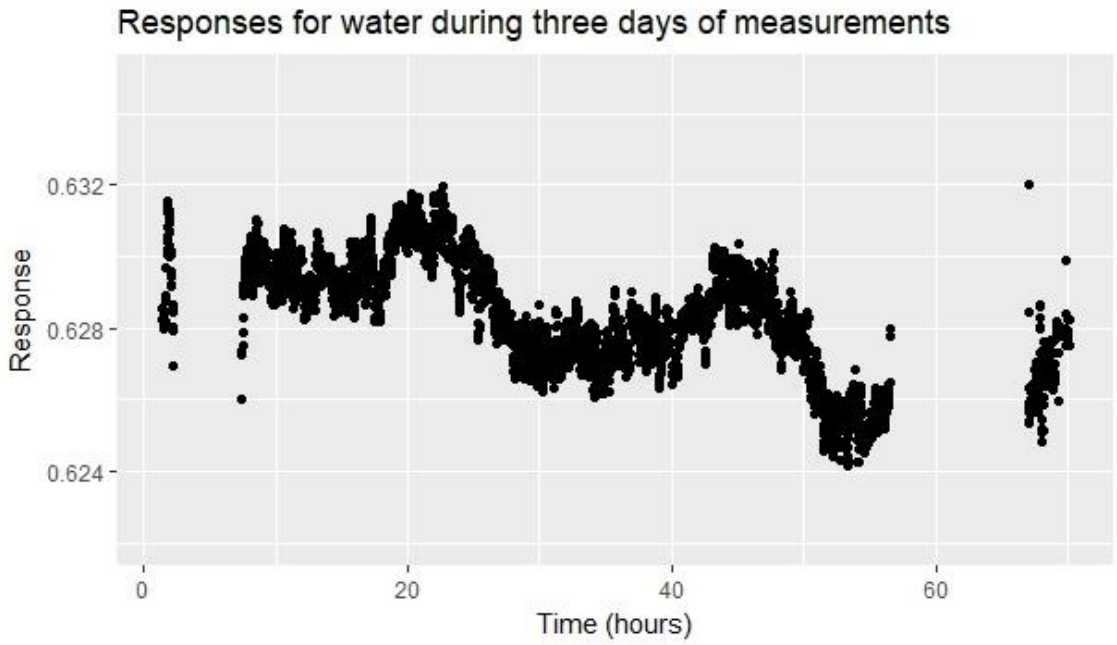

Figure 16. Even with fluctuations, there is declining tendency in responses – correlation between time in hours and response is -0.66.

7 Discussion

7.1 Calibration curves

The calibration curves obtained from the measurements were mostly quite satisfactory, and useful for any practical purposes. It is, therefore, quite safe to assume that the device is quite suitable for monitoring continuous flow of waste or process waters and detecting any abnormal variations. As the device detects change in both quantity and quality of the solids, it can be safely used only when the composition of solids is known, and only concentrations need to be monitored.

The calibration curves should also be drawn separately for different solutions; although in practical use, one device would probably never be used with more than one kind of solution, in one spot.

7.2 Temperature

The temperature of the water affected the response obtained, which is not surprising, as the device works with infrared light. There was a negative correlation between the temperature and the response.

As the temperature of the solution affects the values of the measurements, calibration curves for the solutions should be obtained at the same temperature at which it is used. If there are variations in the temperatures of the water that is monitored, it would be recommendable to draw calibration curves for several temperatures.

7.3 Air bubbles

As was noticed in 5.8, air bubbles in the solution interfere with the infrared light's path through the solution, and cause lower readings. Air bubbles can also occur in solutions naturally, especially if the solution is flowing in a turbid manner. For reliable results, the device should always be placed so that the flow of the liquid is not turbid; not in bends or narrow sections in pipelines.

7.4 Decreasing responses

As was shown in section 6.6, there is some evidence of responses decreasing over time. A common problem for continuously working sensor is fouling. Fouling of the tested sensor would interfere with infrared light detection and hence decrease the recorded values.

This is quite plausible explanation in the case of decreasing baseline values. The sensor was usually only rinsed after use, not washed carefully, so some residues might have accumulated on the sensor's surface. Interestingly, the baseline response peaked after some test runs with clay solution, and it was speculated that clay particles might have scrubbed the sensor clean. The fouling theory was also tested by immersing the sensor in diluted sodium hydroxide solution between measurements with two milk dilution series. Sodium hydroxide solution might have cleaned the sensor, but there was, however, no significant difference between the responses from the test runs before and after the NaOH bath.

Fouling does not, however, explain why the responses also decreased during the three days' continuous measurements with water. It is possible that the lamp in the sensor wears and is not as effective as new.

As the decreasing responses were measured for water and air, it is not possible to conclude that responses necessarily decrease also in normal use with solutions that contain suspended particles. Still, the possibility needs to be taken into account when the sensor is used; the fouling problem, at least, is common for all the sensors.

7.5 Problems in measurements and otherwise: suggestions for further testing

As clay tends to settle down very quickly, magnetic stirrer was used to keep clay from settling and the solution homogenous. Still, with larger concentrations, there was a thin layer of less turbid liquid on the top of the liquid, which means that the mixing was not perfect even with the stirrer.

Similarly, with cement, even stirring was not enough. With the largest concentration, there was a visible layer of clearer solution on the top (see Figure 17). This was, however, partly an artifact caused by the sensor: the layer seemed to disappear when the

sensor was removed and stirring was more efficient. Regardless, the measurements cannot be very accurate in larger concentrations due to settling.

Figure 17. The solids in the cement solution tended to settle despite stirring.

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